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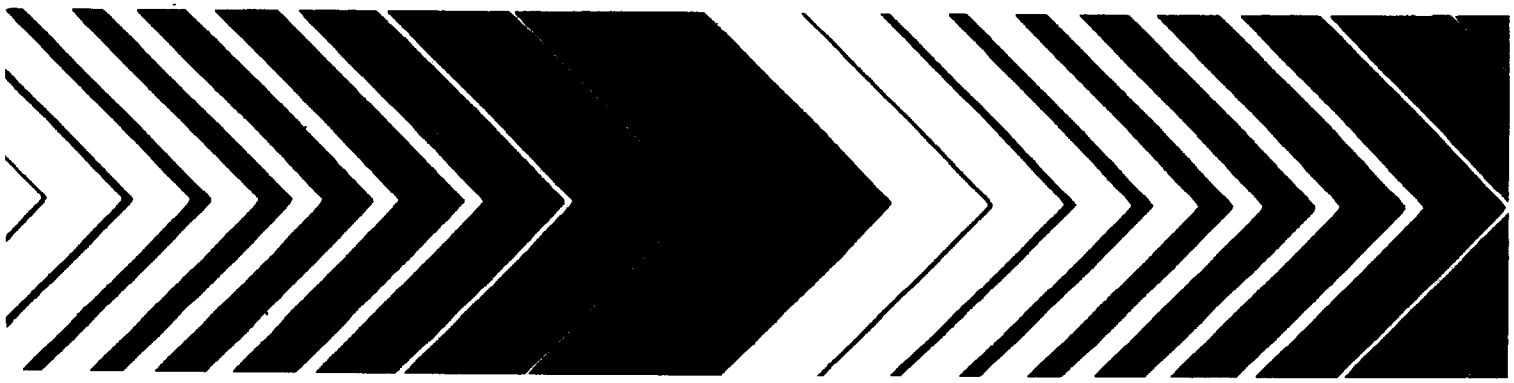
Test Method

Method for the Determination of Asbestos in Bulk Building Materials



EPA/600/R-93/116
July 1993

TEST METHOD

METHOD FOR THE DETERMINATION OF ASBESTOS IN BULK BUILDING MATERIALS

by

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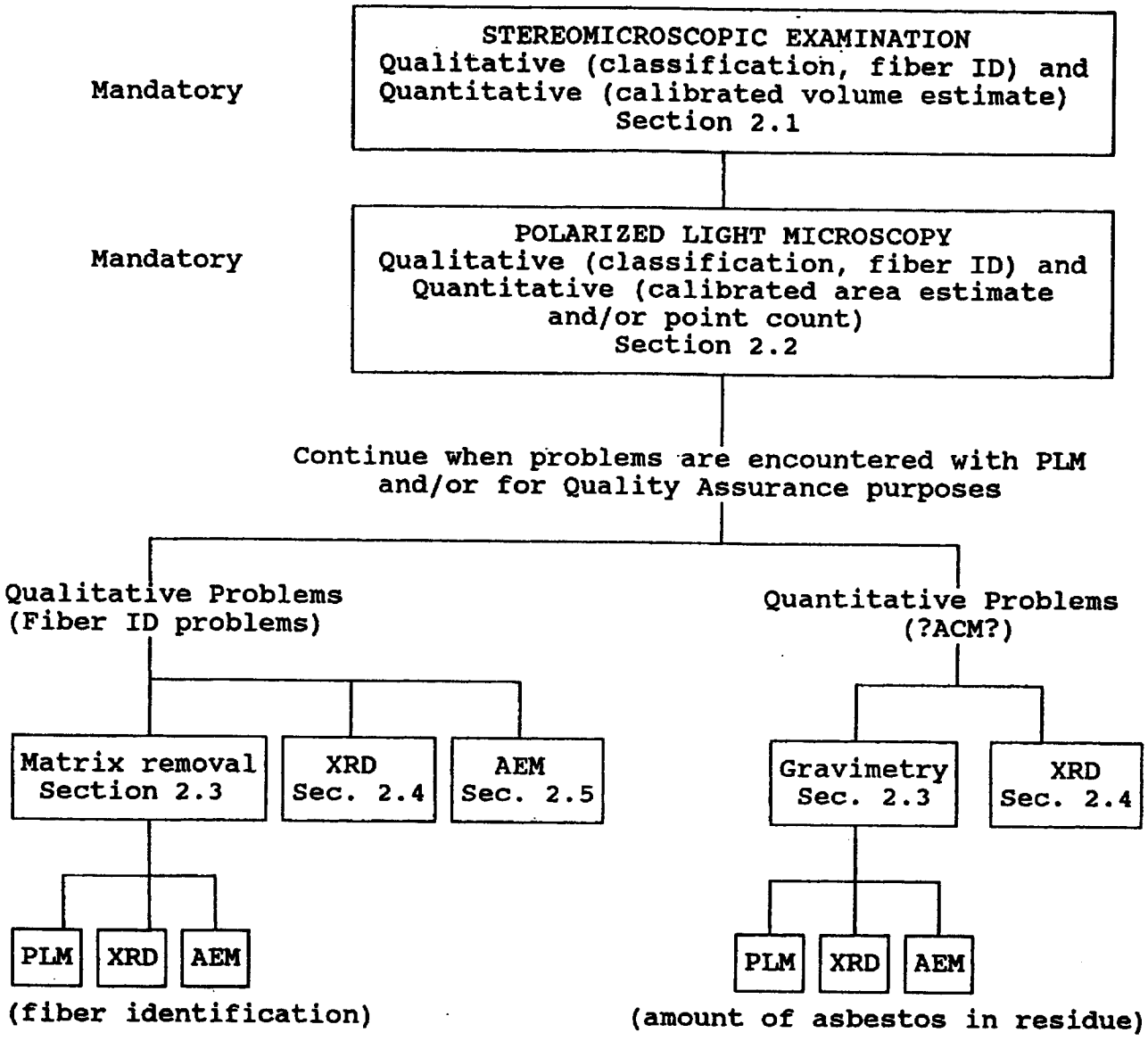
1.0 INTRODUCTION

Laboratories are now called upon to identify asbestos in a variety of bulk building materials, including loose-fill insulations, acoustic and thermal sprays, pipe and boiler wraps, plasters, paints, flooring products, roofing materials and cementitious products.

The diversity of bulk materials necessitates the use of several different methods of sample preparation and analysis. An analysis with a simple stereomicroscope is always followed by a polarized light microscopic (PLM) analysis. The results of these analyses are generally sufficient for identification and quantitation of major concentrations of asbestos. However, during these stereomicroscopic and PLM analyses, it may be found that additional techniques are needed to: 1) attain a positive identification of asbestos; 2) attain a reasonable accuracy for the quantity of asbestos in the sample; or 3) perform quality assurance activities to characterize a laboratory's performance. The additional techniques include x-ray diffraction (XRD), analytical electron microscopy (AEM), and gravimetry, for which there are sections included in the method. Other techniques will be considered by the Environmental Protection Agency (EPA) and may be added at some future time. Table 1-1 presents a simplified flowchart for analysis of bulk materials.

This Method for the Determination of Asbestos in Bulk Building Materials outlines the applicability of the various preparation and analysis methods to the broad spectrum of bulk building materials now being analyzed. This method has been evaluated by the EPA Atmospheric Research and Exposure Assessment Laboratory (EPA/AREAL) to determine if it offers improvements to current analytical techniques for building materials. This method demonstrated a capability for improving the precision and accuracy of analytical results. It contains significant revisions to procedures outlined in the Interim Method,[1] along with the addition of several new procedures. Each technique may reduce or introduce bias, or have some effect on the precision of the measurement, therefore results need to be interpreted judiciously. Data on each technique, especially those new to asbestos analysis, will be collected over time and carefully evaluated, with resulting recommendations for changes to the Method to be passed on to the appropriate program office within EPA.

TABLE 1-1. SIMPLIFIED FLOWCHART FOR ANALYSIS OF BULK MATERIALS



This is an analytical method. It is not intended to cover bulk material sampling, an area addressed previously[2,3,4,5] by the EPA. However, subsampling or sample splitting as it pertains to laboratory analysis procedures in this method, is discussed throughout.

1.1 References

1. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples,** U.S. E.P.A. 600/M4-82-020, 1982.
2. **Asbestos-Containing Materials in School Buildings: A Guidance Document,** Part 1 and 2, U.S. E.P.A./O.T.S NO. C00090, 1979.
3. **Asbestos in Buildings: Simplified Sampling Scheme for Friable Surfacing Materials,** U.S. E.P.A. 560/5-85-030a, 1985.
4. **Guidance for Controlling Asbestos-Containing Materials in Buildings,** U.S. E.P.A. 560/5-85-024, 1985.
5. **Asbestos-Containing Materials in Schools: Final Rule and Notice,** 40 CFR Part 763, October, 1987.

2.0 METHODS

2.1 Stereomicroscopic Examination

A preliminary visual examination using a simple stereomicroscope is mandatory for all samples. A sample should be of sufficient size to provide for an adequate examination. For many samples, observations on homogeneity, preliminary fiber identification and semi-quantitation of constituents can be made at this point. Another method of identification and semi-quantitation of asbestos must be used in conjunction with the stereomicroscopic examination. A description of the suggested apparatus needed for stereomicroscopic examination is given in Appendix B.

The laboratory should note any samples of insufficient volume. A sufficient sample volume is sample-type dependent. For samples such as floor tiles, roofing felts, paper insulation, etc., three to four square inches of the layered material would be a preferred sample size. For materials such as ceiling tiles, loose-fill insulation, pipe insulation, etc., a sample size of approximately one cubic inch ($-$ 15cc) would be preferred. For samples of thin-coating materials such as paints, mastics, spray plasters, tapes, etc., a smaller sample

size may be suitable for analysis. Generally, samples of insufficient volume **should be rejected**, and further analysis curtailed until the client is contacted. The quantity of sample affects the sensitivity of the analysis and reliability of the quantitation steps. If there is a question whether the sample is representative due to inhomogeneity, the sample should be rejected, at least until contacting the client to see if: 1) the client can provide more material or 2) the client wishes the laboratory to go ahead with the analysis, but with the laboratory including a statement on the limited sensitivity and reliability of quantitation. If the latter is the case, the report of analysis should state that the client was contacted, that the client decided that the lab should use less material than recommended by the method, and that the client acknowledges that this may have limited the sensitivity and quantitation of the method. At the time the client is contacted about the material, he or she should be informed that a statement reflecting these facts will be placed in the report.

2.1.1 Applicability

Stereomicroscopic analysis is applicable to all samples, although its use with vinyl floor tile, asphaltic products, etc., may be limited because of small asbestos fiber size and/or the presence of interfering components. It does not provide positive identification of asbestos.

2.1.2 Range

Asbestos may be detected at concentrations less than one percent by volume, but this detection is highly material dependent.

2.1.3 Interferences

Detection of possible asbestos fibers may be made more difficult by the presence of other nonasbestos fibrous components such as cellulose, fiber glass, etc., by binder/matrix materials which may mask or obscure fibrous components, and/or by exposure to conditions (acid environment, high temperature, etc.) capable of altering or transforming asbestos.

2.1.4 Precision and Accuracy

The precision and accuracy of these estimations are material dependent and must be determined by the individual laboratory for the percent range involved. These values may be

determined for an individual analyst by the in-house preparation and analysis of standards and the use of error bars, control charts, etc.

The labs should also compare to National Voluntary Laboratory Accreditation Program (NVLAP) proficiency testing samples, if the lab participates in the Bulk Asbestos NVLAP, or to external quality assurance system consensus results such as from proficiency testing programs using characterized materials. However, at this time, consensus values for the quantity of asbestos have been shown to be unreliable. Only proficiency testing materials characterized by multiple techniques should be used to determine accuracy and precision.

2.1.5 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are oftentimes friable and may release fibers during handling or matrix reduction steps. All sample handling and examination must be carried out in a HEPA-filtered hood, a class 1 biohazard hood or a glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst to and contamination of samples by airborne fibers.

2.1.5.1 Sample Preparation

No sample preparation should be undertaken before initial stereomicroscopic examination. Distinct changes in texture or color on a stereomicroscopic scale that might denote an uneven distribution of components should be noted. When a sample consists of two or more distinct layers or building materials, each should be treated as a separate sample, when possible. Thin coatings of paint, rust, mastic, etc., that cannot be separated from the sample without compromising the layer are an exception to this case and may be included with the layer to which they are attached. Drying (by heat lamp, warm plate, etc.) of wet or damp samples is recommended before further stereomicroscopic examination and is mandatory before PLM examination. **Drying must be done in a safety hood.**

For nonlayered materials that are heterogeneous, homogenization by some means (mill, blender, mortar and pestle) may provide a more even distribution of sample components. It

may also facilitate disaggregation of clumps and removal of binder from fibers (rarely however, it may mask fibers that were originally discernable).

For materials such as cementitious products and floor tiles, breaking, pulverizing, or grinding may improve the likelihood of exposing fibrous components.

It may be appropriate to treat some materials by dissolution with hydrochloric acid to remove binder/matrix materials. Components such as calcite, gypsum, magnesite, etc., may be removed by this method. For materials found to possess a high organic content (cellulose, organic binders), ashing by means of a muffle furnace or plasma asher (for small, cellulosic samples), or dissolution by solvents may be used to remove interfering material. In either case, it is recommended that matrix removal be tracked gravimetrically.

Additional information concerning homogenization, ashing and acid dissolution may be found in Sections 2.2.5.1 and 2.3.

2.1.5.2 Analysis

Samples should be examined with a simple stereomicroscope by viewing multiple fields of view over the entire sample. The whole sample should be observed after placement in a suitable container (watchglass, weigh boat, etc.) substrate. Samples that are very large should be subsampled. The sample should be probed, by turning pieces over and breaking open large clumps. The purpose of the stereomicroscopic analysis is to determine homogeneity, texture, friability, color, and the extent of fibrous components of the sample. This information should then be used as a guide to the selection of further, more definitive qualitative and quantitative asbestos analysis methods. Homogeneity refers to whether each subsample made for other analytical techniques (e.g. the "pinch" mount used for the PLM analysis), is likely to be similar or dissimilar. Color can be used to help determine homogeneity, whether the sample has become wet (rust color), and to help identify or clarify sample labelling confusion between the building material sampler and the laboratory. Texture refers to size, shape and arrangement of sample components. Friability may be indicated by the ease with which the sample is disaggregated (see definitions in Appendix A) as received by the analyst. This does not necessarily represent the friability of the material as determined by the assessor at the collection site. The relative proportion of fibrous

components to binder/matrix material may be determined by comparison to similar materials of known fibrous content. For materials composed of distinct layers or two or more distinct building materials, each layer or distinct building material should be treated as a discrete sample. The relative proportion of each in the sample should be recorded. The layers or materials should then be separated and analyzed individually. Analysis results for each layer or distinct building material should be reported. If monitoring requirements call for one reported value, the results for the individual layers or materials should always be reported along with the combined value. Each layer or material should be checked for homogeneity during the stereomicroscopic analysis to determine the extent of sample preparation and homogenization necessary for successful PLM or other analysis. Fibers and other components should be removed for further qualitative PLM examination.

Using the information from the stereomicroscopic examination, selection of additional preparation and analytical procedures should be made. Stereomicroscopic examination should typically be performed again after any change or major preparation (ashing, acid dissolution, milling, etc.) to the sample. Stereomicroscopic examination for estimation of asbestos content may also be performed again after the qualitative techniques have clarified the identities of the various fibrous components to assist in resolving differences between the initial quantitative estimates made during the stereomicroscopic analysis and those of subsequent techniques. Calibration of analysts by use of materials of known asbestos content is essential.

The stereomicroscopic examination is often an iterative process. Initial examination and estimates of asbestos concentration should be made. The sample should then be analyzed by PLM and possibly other techniques. These results should be compared to the initial stereomicroscopic results. Where necessary, disagreements between results of the techniques should be resolved by reanalyzing the sample stereomicroscopically.

2.1.6 Calibration Materials

Calibration materials fall into several categories, including internal laboratory standards and other materials that have known asbestos weight percent content. These calibration materials could include:

- Actual bulk samples: asbestos-containing materials that have been characterized by other analytical methods such as XRD, AEM and/or gravimetry. (e.g. NVLAP test samples).

- Generated samples: in-house standards that can be prepared by mixing known quantities of asbestos and known quantities of asbestos-free matrix materials (by weight), and mixing (using blender, mill, etc.) thoroughly to achieve homogeneity; matrix materials such as vermiculite, perlite, sand, fiberglass, calcium carbonate, etc. may be used. A range of asbestos concentrations should be prepared (e.g. 1, 3, 5, 10, 20%, etc.). The relationship between specific gravities of the components used in standards should be considered so that weight/volume relationships may be determined.

- Photographs, drawings: photomicrographs of standards, computer-generated drawings, etc.

Suggested techniques for the preparation and use of in-house calibration standards are presented in Appendix C, and at greater length by Harvey et al.[1] The use of synthesized standards for analyst calibration and internal laboratory quality control is not new however, having been outlined by Webber et al.[2] in 1982.

2.1.7 References

1. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991, pp. 22-29.

2. Webber, J. S., A. Pupons and J. M. Fleser, "Quality-Control Testing for Asbestos Analysis with Synthetic Bulk Materials". **American Industrial Hygiene Associations Journal**, 43, 1982, pp. 427-431.

2.2 Polarized Light Microscopy

2.2.1 Principle and Applicability

Samples of bulk building materials taken for asbestos identification should first be examined with the simple stereomicroscope to determine homogeneity and preliminary fiber identification. Subsamples should then be examined using PLM to determine optical properties of constituents and to provide positive identification of suspect fibers.

The principles of optical mineralogy are well-established.[1,2,3,4] A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows for the determination of refractive indices relative to specific crystallographic orientations. Morphology and color are also observed while viewing under plane polarized light. Observation of particles or fibers while oriented between polarizing filters whose privileged vibration directions are perpendicular (crossed polars) allows for determination of isotropism/anisotropism, extinction characteristics of anisotropic particles, and calculation of birefringence. A retardation plate may be placed in the polarized light path for verification of the sign of elongation. If subsamples are prepared in such a way as to represent all sample components and not just suspect fibers, semi-quantitative analysis may also be performed. Semi-quantitative analysis involves the use of calibrated visual area estimation and/or point counting. Visual area estimation is a semi-quantitative method that must relate back to calibration materials. Point counting, also semi-quantitative, is a standard technique used in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting[3] and the interpretation of point count data[5] is available.

Although PLM analysis is the primary technique used for asbestos determination, it can show significant bias leading to false negatives and false positives for certain types of materials. PLM is limited by the visibility of the asbestos fibers. In some samples the fibers may be reduced to a diameter so small or masked by coatings to such an extent that they cannot be reliably observed or identified using PLM.

2.2.2 Range

The detection limit for visual estimation is a function of the quantity of sample analyzed, the nature of matrix interference, sample preparation, and fiber size and distribution. Asbestos may be detected in concentrations of less than one percent by area if sufficient material is analyzed. Since floor tiles may contain fibers too small to be resolved by PLM ($< 0.25\ \mu m$ in diameter), detection of those fibers by this method may not be possible. When point counting is used, the detection limit is directly proportional to the amount of sample analyzed, but is also limited by fiber visibility. Quantitation by area estimation, both visual and by point counting, should yield similar results if based on calibration standards.

2.2.3 Interferences

Fibrous and nonfibrous, organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Binder/matrix materials may coat fibers, affect color, or obscure optical characteristics to the extent of masking fiber identity. Many organic mastics are soluble in refractive index liquids and, unless removed prior to PLM examination, may affect the refractive index measurement of constituent materials. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification. Gravimetric procedures for the removal of interfering materials are presented in Section 2.3.

2.2.4 Precision and Accuracy

Data obtained for samples containing a single asbestos type in a sample matrix have been reported previously by Brantley et al.[6] Data for establishing the accuracy and precision of the method for samples with various matrices have recently become available. Perkins,[7] Webber et al.[8] and Harvey et al.[9] have each documented the tendency for visual estimates to be high when compared to point-count data. Precision and accuracy must be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonably expanded errors, such as those shown in Table 2-1, should be generated for different concentrations of asbestos.

If the laboratory cannot demonstrate adequate precision and accuracy (documented by control charts, etc), quantitation by additional methods, such as gravimetry, may be required. Refer to the Handbook for SRM Users[10] for additional information concerning the concepts of precision and accuracy.

TABLE 2-1. SUGGESTED ACCEPTABLE ERRORS FOR PLM ANALYSIS
(Based on 400 point counts of a reasonably homogeneous sample or 100 fields of view for visual estimate)

% Area Asbestos	Acceptable Mean Result	% Area Asbestos	Acceptable Mean Result
1	>0-3%	50	40-60%
5	>1-9%	60	50-70%
10	5-15%	70	60-80%
20	10-30%	80	70-90%
30	20-40%	90	80-100%
40	30-50%	100	90-100%

2.2.5 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are oftentimes friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations must be carried out in a HEPA-filtered, a class 1 biohazard hood, or a glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst to and contamination of samples by airborne fibers.

2.2.5.1 Sample Preparation

Slide mounts are prepared for the identification and quantitation of asbestos in the sample.

2.2.5.1.1 Qualitative Analysis Preparation

The qualitative preparation must allow the PLM analysis to classify the fibrous components of the sample as asbestos or nonasbestos. The major goal of the qualitative

preparation is to mount easily visible fibers in appropriate refractive index liquids for complete optical characterization. Often this can be accomplished by making immersion grain mounts of random subsamples of the homogeneous material. Immersion liquids with refractive indices close to the suspected (see stereomicroscopic analysis) asbestos mineral should be used for the qualitative analysis so that n_D can be determined. Problem samples include those with inhomogeneities, coatings, small fibers, and interfering compounds. Additional qualitative preparations are often necessary for these types of samples. All samples, but especially those lacking homogeneity, may require picking of fibers from specific sample areas during the stereomicroscopic examination. Coatings on the fibers often need to be removed by mechanical or chemical means. Teasing the particles apart or use of a mortar and pestle or similar mechanical method often is sufficient to free fibers from coatings. Chemical means of removing some coatings and interfering compounds are discussed in Section 2.3, Gravimetry.

2.2.5.1.2 Quantitative Analysis Preparation

The major purpose of the quantitative preparation is to provide the analyst with a representative grain mount of the sample in which the asbestos can be observed and distinguished from the nonasbestos matrix. This is typically performed by using randomly selected subsamples from a homogeneous sample (see stereomicroscopic analysis). Particles should be mounted in a refractive index (RI) liquid that allows the asbestos to be visible and distinguished from nonasbestos components. Care should be taken to ensure proper loading and even distribution of particles. Both the qualitative and quantitative sample preparations are often iterative processes. Initial samples are prepared and analyzed. The PLM analysis may disclose problems or raise questions that can only be resolved by further preparations (e.g. through the use of different RI immersion liquids, elimination of interfering compounds, sample homogenization, etc.)

For layered materials, subsamples should be taken from each individual or discrete layer. Each of these subsamples should be treated as a discrete sample, but as stated in Section 2.1.5.2, the results for the individual layers or materials may be combined if called for by monitoring requirements.

Homogenization involves the use of any of a variety of devices, such as a mortar and pestle, mill, or blender to pulverize, disaggregate and mix heterogeneous, friable bulk materials. Selection of the appropriate device is dependent upon personal preference and the nature of the materials encountered. A blender or mortar and pestle may be adequate for homogenizing materials that lack appreciable amounts of tacky matrix/binder, and for separating interfering components from the fibers. For materials which are unusually sticky or tacky, or contain unusually long asbestos fibers, milling (especially freezer milling) may be more efficient. However, milling should be discontinued as soon as the material being milled appears homogeneous, in order to reduce the potential for mechanically reducing fiber size below the resolving power of the polarizing microscope. Hammer mills or cutting mills may also be used on these materials; however, the same precaution regarding reduction of fiber size should be taken. Blending /milling devices should be disassembled (to the extent possible) and thoroughly cleaned after each use to minimize contamination.

2.2.5.2 Analysis

Analysis of bulk building materials consists of the identification and semi-quantitation of the asbestos type(s) present, along with the identification, where possible, of fibrous nonasbestos materials, mineral components and matrix materials. If the sample is heterogeneous due to the presence of discrete layers or two or more distinct building materials, each layer or distinct material should be analyzed, and results reported. Total asbestos content may also be stated in terms of a relative percentage of the total sample.

2.2.5.2.1 Identification

Positive identification of asbestos requires the determination of the following optical properties:

- Morphology
- Color and, if present, pleochroism
- Refractive indices (± .005)
- Birefringence
- Extinction characteristics
- Sign of elongation

Descriptions of the optical properties listed above for asbestos fibers may be found in Appendix A, Glossary of Terms. Table 2-2 lists the above properties for the six types of asbestos and Table 2-3 presents the central stop dispersion staining colors for the asbestos minerals with selected high-dispersion index liquids. Tables 2-4 and 2-5 list selected optical properties of several mineral and man-made fibers. All fibrous materials in amounts greater than trace should be identified as asbestos or nonasbestos, with all optical properties measured for asbestos and at least one optical property measured for each nonasbestos fibrous component that will distinguish each from asbestos. Small fiber size and/or binder may necessitate viewing the sample at higher magnification (400-500x) than routinely used (100x).

Although it is not the purpose of this section to explain the principles of optical mineralogy, some discussion of the determination of refractive indices is warranted due to its importance to the proper identification of the asbestos minerals. Following is a brief discussion of refractive index determination for the asbestos minerals.

All asbestos minerals are anisotropic, meaning that they exhibit different optical properties (including indices of refraction) in different directions. All asbestos minerals are biaxial, meaning that they have one principal refractive index parallel (or nearly parallel) to the length of the fiber and two principal refractive indices (plus all intermediate indices between these two) in the plane perpendicular (or nearly so) to the length of the fiber. Although chrysotile (serpentine) is classified as a biaxial mineral, it behaves as a uniaxial mineral (two principal refractive indices) due to its scrolled structure. Amosite and crocidolite, although also biaxial, exhibit uniaxial properties due to twinning of the crystal structure and/or random orientation of fibrils in a bundle around the long axis of the bundle. For all of the asbestos minerals except crocidolite, the highest refractive index (γ) is aligned with the fiber length (positive sign of elongation). For crocidolite, the lowest refractive index (α) is aligned with the fiber length (negative sign of elongation). A more complete explanation of the relationship of refractive indices to the crystallographic directions of the asbestos minerals may be found in References 1, 2, 4, 11 and 12. It should be noted that for the measurement of refractive indices in an anisotropic particle (e.g. asbestos fibers), the orientation of the particle is quite critical. Orientation with respect to rotation about the axis

of the microscope (and thus with respect to the vibration directions of the polarizer and analyzer) and also to the horizontal plane (plane of the microscope stage) will affect the determination of the correct values for refractive indices. The refractive index that is measured will always correspond to a direction perpendicular to the axis of the microscope (i.e., lying in the plane of the stage) and is the direction in that horizontal plane parallel to the vibration direction of the polarizer, by convention E-W.

To determine $\gamma(n\parallel)$ for chrysotile, anthophyllite and amosite, the index is measured when the length of the fiber is aligned parallel to the vibration direction of the polarizer (E-W). Under crossed polars, the fiber should be at extinction in this orientation. To determine the lowest refractive index, α ($n\perp$), for chrysotile and amosite, the fiber should be oriented N-S (extinction position under crossed polars). The determination of $n\parallel$ and $n\perp$ with crocidolite is accomplished in the same manner as with amosite and chrysotile with the exception that the α and γ directions are reversed. For crocidolite, α is measured at the E-W position (parallel to the polarizer) and γ is measured at the N-S orientation (perpendicular to the polarizer). For anthophyllite, the fiber should be oriented N-S and the lowest and highest indices for this orientation should be measured. These correspond to α and β respectively.

The extinction behavior of tremolite-actinolite is anomalous compared to that of most monoclinic minerals due to the orientation of the optic axes relative to the crystallographic axes. This relationship is such that the refractive indices of the principal axes α and γ <u>are not measured</u> when the fiber is exhibiting the maximum extinction angle. The values measured at these positions are α' and γ'. The fiber exhibits an extinction angle within a few degrees of the maximum throughout most of its rotation. A wide range of refractive indices from α' to α, and from γ' to γ, are observed. For tremolite-actinolite, β is measured on those fibers displaying parallel extinction when oriented in the N-S position. The refractive index for α is also measured when the fiber is oriented generally in the N-S position and exhibits <u>the true extinction</u> angle; true α will be the <u>minimum</u> index. To determine the refractive index for γ, the fibers should be oriented E-W and exhibit the true extinction angle; true γ will be the <u>maximum</u> value for this orientation.

When viewing single fibers, the analyst may often be able to manipulate the microscope slide cover slip and "roll" the fibers to positions that facilitate measuring the true values of refractive indices. When viewing a large population of fibers with the microscope in the dispersion staining mode, the analyst can easily detect fibers that exhibit the highest and lowest indices (β and α) in the N-S position and the highest indices (γ) in the E-W position. Since individual asbestos fibrils cannot generally be resolved using polarized light microscopy, refractive indices are most commonly measured on fiber bundles. Such measurements would not result in true values for the indices and therefore by convention should be reported as α' and γ'.

Asbestos types chrysotile, amosite and crocidolite are currently available as SRM 1866 and actinolite, tremolite and anthophyllite as SRM 1867 from the Office of Standard Reference Materials, National Institute of Standards and Technology.

2.2.5.2.2 Quantitation of Asbestos Content

As described in Sections 2.1.5 and 2.1.6, a calibrated visual volume estimation of the relative concentrations of asbestos and nonasbestos components should be made during the stereomicroscopic examination. In addition, quantitation of asbestos content should be performed on subsample slide mounts using calibrated visual area estimates and/or a point counting procedure. Section 2.1.6 and Appendix C discuss the procedures for preparation and use of calibration standards. After thorough PLM analysis in which the asbestos and other components of the bulk material are identified, several slides should be carefully prepared from randomly selected subsamples. If the sample is not homogeneous, some homogenization procedure should be performed to ensure that slide preparations made from small pinch samples are representative of the total sample. Homogenization may range from gentle mixing using a mortar and pestle to a brief period of mixing using a blender equipped with a mini-sample container. The homogenization should be of short duration ($\sim$ 15 seconds) if using the blender technique so as to preclude a significant reduction in fiber size. The use of large cover slips (22x30mm) allows for large subsamples to be analyzed. Each slide should be checked to ensure that the subsample is representative, uniformly dispersed, and loaded in a way so as not to be dominated by superimposed (overlapping) particles.

During the qualitative analysis of the sample, the analyst should decide on the appropriate optical system (including magnification) to maximize the visibility of the asbestos in the sample while still allowing the asbestos to be uniquely distinguished from the matrix materials. The analyst may choose to alter the mounting medium or the optical system to enhance contrast. During the quantitative analysis, slides should be scanned using an optical setup that yields the best visibility of the asbestos. Upon finding asbestos, the parameters that were selected in the qualitative analysis for uniquely distinguishing it from the matrix should be used for identification. These properties will vary with the sample but include any or all of the parameters required for the qualitative analysis. For instance, low magnification allows for concurrent use of dispersion staining (focal screening), but compromises resolution of extremely small diameter fibers; use of a compensator plate and crossed polarizers frequently enhances the contrast between asbestos fibers and matrix material.

Visual area estimates should be made by comparison of the sample to calibration materials that have similar textures and fiber abundance (see Section 2.1.6 and Appendix C). A minimum of three slide mounts should be examined to determine the asbestos content by visual area estimation. Each slide should be scanned in its entirety and the relative proportions of asbestos and nonasbestos noted. It is suggested that the ratio of asbestos to nonasbestos material be recorded for several fields for each slide and the results be compared to data derived from the analysis of calibration materials having similar textures and asbestos content.

For point counting, an ocular reticle (cross-line or point array) should be used to visually superimpose a point or points on the microscope field of view. The cross-line reticle is preferred. Its use requires the scanning of most, if not all, of the slide area, thereby minimizing bias that might result from lack of homogeneity in the slide preparation. In conjunction with this reticle, a click-stop counting stage can be used to preclude introducing bias during slide advancement. Magnification used will be dictated by fiber visibility. The slide should be examined along multiple parallel traverses that adequately cover the sample area. The analyst should score (count) only points directly over occupied (nonempty) areas. Empty points should not be scored on the basis of the closest particle. If an asbestos fiber and a nonasbestos particle overlap so that a point is superimposed on their visual intersection,

a point should be scored for both categories. If the point(s) is/are superimposed on an area which has several overlapping particles, the slide should be moved to another field. While not including them in the total asbestos points counted, the analyst should record the presence of any asbestos detected but not lying under the reticle cross-line or array points. A minimum of 400 counts (maximum of eight slides with 50 counts each to minimum of two slides with 200 counts each) per sample is suggested, but it should be noted that accuracy and precision improve with number of counts. Point counting provides a determination of the projected area percent asbestos. Conversion of area percent to dry weight percent is not feasible unless the specific gravities and relative volumes of the different materials are known. It should be noted that the total amount of material to be analyzed is dependent on the asbestos concentration, i.e. the lower the concentration of asbestos, the larger the amount of sample that should be analyzed, in both the visual estimation and point counting methods. Quantitation by either method is made more difficult by low asbestos concentration, small fiber size, and presence of interfering materials.

It is suggested that asbestos concentration be reported as volume percent, weight percent or area percent depending on the method of quantitation used. A weight concentration cannot be determined without knowing the relative specific gravities and volumes of the sample components.

TABLE 2-2. OPTICAL PROPERTIES OF ASBESTOS FIBERS

Mineral	Morphology and Color[1]	Refractive Indices[2] α	Refractive Indices[2] γ[5]	Birefringence[6]	Extinction	Sign of Elongation
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1. Colorless[3]	1.493-1.546 1.532-1.549 1.529-1.559 1.544-1.553	1.517-1.557 1.545-1.556 1.537-1.567 1.552-1.561	0.004-0.017	Parallel	+ (length slow)
Amosite (asbestiform grunerite)	Straight to curved, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so.[4] Opaque inclusions may be present	1.657-1.663 1.663-1.686 1.663-1.686 1.676-1.683	1.699-1.717 1.696-1.729 1.696-1.729 1.697-1.704	0.021-0.054	Usually parallel	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight to curved, rigid fibers. Aspect ratio typically > 10:1. Thick fibers and bundles common, blue to dark-blue in color. Pleochroic.	1.693 1.654-1.701 1.680-1.698	1.697 1.668-1.717 1.685-1.706	0.003-0.022	Usually parallel	- (length fast)
Anthophyllite-asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Anthophyllite cleavage fragments may be present with aspect ratios <10:1. Colorless to light brown.	1.598-1.652 1.596-1.694 1.598-1.674 1.6148[7]	1.623-1.676 1.615-1.722 1.615-1.697 1.6362[7]	0.013-0.028	Parallel	+ (length slow)
Tremolite-Actinolite-asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Cleavage fragments may be present with aspect ratios <10:1. Colorless to pale green	Tremolite 1.600-1.628 1.604-1.612 1.599-1.612 1.6063[7]	 1.625-1.655 1.627-1.635 1.625-1.637 1.6343[7]	 0.017-0.028	Parallel and oblique (up to 21°); Composite fibers show parallel extinction.	+ (length slow)
		Actinolite 1.600-1.628 1.612-1.668 1.613-1.628 1.6126[7]	 1.625-1.655 1.635-1.688 1.638-1.655 1.6393[7]	 0.017-0.028		

[1]Colors cited are seen by observation with plane polarized light.

[2]From references 2, 11, 12, and 18, respectively. Refractive indices for n_d at 589.3nm.

[3]Fibers subjected to heating may be brownish. (references 13, 14, and 15)

[4]Fibers subjected to heating may be dark brown and pleochroic. (references 13, 14, and 15)

[5]∥ to fiber length, except ⊥ to fiber length for crocidolite only.

[6]Maximum and minimum values from references 2, 11, 12, and 18 given.

[7]± 0.0007

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TABLE 2-3. TYPICAL CENTRAL STOP DISPERSION STAINING COLORS[1]

Mineral	Cargille® RI Liquid	n‖	n⊥
Chrysotile	1.550HD	Magenta to light blue-green λ_0's ca. 520-620nm	Blue-green to pale blue λ_0's ca. 600-700nm
Amosite	1.680	Yellow to magenta λ_0's ca. 420-520nm	Blue magenta to light blue λ_0's ca. 560-660nm
Crocidolite	1.680	Yellow to magenta λ_0's ca. 420-520nm	Pale yellow to golden yellow λ_0's ca. 360-460nm
Anthophyllite-asbestos	1.605HD	Pale yellow to yellow λ_0's ca. 330-430nm	Golden yellow to light blue green λ_0's ca. 460-700nm
Tremolite-asbestos	1.605HD	Pale yellow to yellow λ_0's ca. 330-430nm	Golden yellow to light blue green λ_0's ca. 460-700nm
Actinolite-asbestos	1.605HD	Pale yellow λ_0's ca. 260-360nm	Pale yellow to golden yellow λ_0's ca. 360-460nm
	1.630HD	Yellow to magenta λ_0's ca. 420-520nm	Golden yellow to blue λ_0's ca. 450-600nm

[1]Modified from reference 16

TABLE 2-4. OPTICAL PROPERTIES OF MAN-MADE TEXTILE FIBERS[1,2]

Fiber Type	n‖	n⊥	n‖ - n⊥	Sign of Elongation
Polyester (Dacron®)	1.710	1.535	0.175	+
Polyamide (Nylon®)	1.582	1.514	0.063	+
Aramid (Kevlar®)	≈2.37	≈1.641	0.729	+
Olefin (Polyethylene)	1.556	1.512	0.044	+
Olefin (Polypropylene)	1.520	1.495	0.025	+
Viscose Rayon	1.535-1.555	1.515-1.535	0.020	+
Acetate	1.478-1.480	1.473-1.476	0.004-0.005	+
Acrylic (Orlon®)	1.505-1.515	1.507-1.517	0.004-0.002	-
Modacrylic (Dynel®)	1.535	1.532	0.002	+

[1]Modified from reference 17

[2]Refractive indices for specific fibers; other fibers may vary

TABLE 2-5. OPTICAL PROPERTIES OF SELECTED FIBERS[1]

FIBER TYPE	MORPHOLOGY	REFRACTIVE INDICES	BIREFRINGENCE ($n\parallel - n\perp$)	EXTINCTION ANGLE	SIGN OF ELONGATION	DISPERSION STAINING COLORS
Paper (Cellulose)	Tapered, flat ribbons	$n\parallel \sim 1.580$ $n\perp \sim 1.530$	High (0.05)	Parallel and incomplete	+	in 1.550HD $n\parallel$: yellow (λ_o's < 400nm) $n\perp$: pale blue (λ_o's > 700nm)
Olefin (polyethylene)	Filaments or shredded like chrysotile	$n\parallel \sim 1.556$ $n\perp \sim 1.512$	Moderate (0.044)	Parallel	+	in 1.550HD $n\parallel$: yellow to magenta (λ_o's = 440-540nm) $n\perp$: pale blue (λ_o's > 700nm)
Brucite (nemalite)	Straight fibers	$n\parallel \sim 1.560$-1.590 $n\perp \sim 1.580$-1.600	Moderate (0.012-0.020)	Usually parallel	- occasionally +	in 1.550HD $n\parallel$: golden yellow (λ_o's 440-460nm) $n\perp$: yellow (λ_o's 400-440nm)
Heated amosite	Similar to unheated, (brittle and shorter) pleochroic: $n\parallel$-dark brown $n\perp$ yellow	$n\parallel$ and $n\perp > 1.700$[2]	High (> 0.05)	Usually parallel	+	in 1.680HD $n\parallel$ & $n\perp$: both pale yellow to white (λ_o's < 400nm)
Glass fibers. Mineral wool	Exotic shapes, tear drops, single filaments	1.515-1.700	Isotropic	------	-	in 1.550HD usually pale blue to blue (λ_o's 580 to > 700nm)
Wollastonite	Straight needles and blades	$n\parallel \sim 1.630$ $n\perp \sim 1.632$ $n\perp$ also 1.610	Moderate to low (0.018 to 0.002)	Parallel and oblique	+ and -	in 1.605HD $n\parallel$ & $n\perp$: yellow to pale yellow (λ_o's < 460nm)
Fibrous talc	Thin cleavage ribbons and wavy fibers	$n\parallel \sim 1.60$ $n\perp \sim 1.54$	High (0.06)	Parallel and oblique	+	in 1.550HD $n\parallel$: pale yellow (λ_o's <400nm) $n\perp$: pale blue (λ_o's >660nm)

[1]From reference 19

[2]From references 13. 14. and 15

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2.2.5.2.3 Microscope Alignment

In order to accurately measure the required optical properties, a properly aligned polarized light microscope must be utilized. The microscope is aligned when:

1) the privileged directions of the substage polarizer and the analyzer are at 90° to one another and are represented by the ocular cross-lines;

2) the compensator plate's privileged vibration directions are 45° to the privileged directions of the polarizer and analyzer;

3) the objectives are centered with respect to stage rotation; and,

4) the substage condenser and iris diaphragm are centered in the optic axis.

Additionally, the accurate measurement of the refractive index of a substance requires the use of calibrated refractive index liquids. These liquids should be calibrated regularly to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or R.I. glass beads.

2.2.6 References

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2.3 Gravimetry

2.3.1 Principle and Applicability

Many components of bulk building materials, specifically binder components, can be selectively removed using appropriate solvents or, in the case of some organics, by ashing. The removal of these components serves the following purposes:

1) to isolate asbestos from the sample, allowing its weight to be determined;

2) to concentrate asbestos and therefore lower the detection limit in the total sample;

3) to aid in the detection and identification of fibrous components; and,

4) to remove organic (ashable) fibers which are optically similar to asbestos.

Common binder materials which are removed easily using the techniques described include: 1) calcite, gypsum, magnesite, brucite, bassanite, portlandite, and dolomite, using hydrochloric acid, and 2) vinyl, cellulose, and other organic components, by ashing. The removal of the binder components results in a residue containing asbestos, if initially present, and any other non-soluble or non-ashable components which were present in the original sample. Unless the procedures employed result in the loss of some asbestos, the weight percent of the residue is the upper limit for the weight percent of asbestos in the sample.

This section describes the procedure for removing acid-soluble and ashable components, and for determining the weight percent of the residue. However, the acid dissolution and ashing techniques can be used without the accompanying weight measurements to either liberate or clean fibers to aid in qualitative PLM or AEM analyses.

This technique is not an identification technique. Other methods, such as PLM, XRD, or AEM must be used to determine the identity of the components. A description of the suggested apparatus, reagents, etc. needed for the techniques described is included in Appendix B.

2.3.2 Interferences

Any components which cannot by removed from the sample by selective dissolution or ashing interfere with asbestos quantitation. These components include, but are not limited to, many silicates (micas, glass fibers, etc.) and oxides (TiO_2, magnetite, etc.). When interfering phases are present (the residue contains other phases in addition to asbestos), other techniques such as PLM, AEM, or XRD must be used to determine the percent of asbestos in the residue.

Care must be taken to prevent loss of or chemical/structural changes in the critical components (asbestos). Prolonged exposure to acids or excessive heating (above 500°C) can cause changes in the asbestos components in the sample and affect the optical properties.[1,2,3]

2.3.3 Quantitation

The weight of the residue remaining after solvent dissolution/ashing should be compared with the original weight of the material. Presuming no insoluble material is lost, the weight percent of the residue is the upper limit for the amount of asbestos in the sample. If the residue is comprised only of asbestos, then the weight percent of residue equals the weight percent of asbestos in the sample. If the residue contains other phases, then techniques such as PLM, XRD, or AEM must be employed to determine the relative abundance of asbestos in the residue.

The precision and accuracy of the technique are dependent upon the homogeneity of the material, the accuracy of the weight measurements, and the effectiveness of the sample reduction and filtering procedures. In practice, the precision can be equal to $\pm 1\%$, and the accuracy at 1 wt% asbestos can be less than or equal to $\pm 10\%$ relative.

The incomplete solution of components and the presence of other nonasbestos components in the residue contribute to producing a positive bias for the technique (falsely high percentages of asbestos).

2.3.4 Preliminary Examination and Evaluation

Stereomicroscopic and PLM examinations of the sample should already have been conducted prior to initiating this procedure. These examinations should have provided information about: 1) whether the sample contains components which can be removed by acid-washing, solvent dissolution, or ashing, and 2) whether the sample contains asbestos, or fibers that might be asbestos, or whether no asbestos was detected.

If the sample is friable and contains organic (ashable) components, the ashing procedure should be followed. If the sample is friable and contains HCl-soluble components, the acid dissolution procedure should be followed. If the sample is friable and contains both types of

components, the two procedures can be applied, preferably with acid dissolution following ashing.

If the sample is nonfriable (e.g. floor tiles), it is also recommended that the ashing procedure be used first, followed by the acid dissolution procedure. The ashing procedure reduces floor tiles to a material which is easily powdered, simplifying the sample preparation for acid dissolution.

2.3.5 Sample Preparation

2.3.5.1 Drying

Any moisture in the sample will affect the weight measurements, producing falsely low percentages of residue. If the sample is obviously wet, it should be dried at low temperature (using a heat lamp, or simply by exposure at ambient conditions, prior to starting the weighing procedure). If an oven is used, the drying temperature should not exceed 60°C. Drying by means of heat lamp or ambient air must be performed within a safety-filtered hood. Even if the sample appears dry, it can contain enough moisture to affect the precision and accuracy of the technique. The test for sample moisture involves placing the amount of sample to be used on the weighing pan; if the weight remains stable with time, then the sample is dry enough. If the weight decreases as the sample sits on the weighing pan, then the sample should be dried. Where conditions of moderate to high humidity are known to exist, all materials to be weighed should be allowed time to stabilize to these ambient conditions.

2.3.5.2 Homogenization/Grain Size Reduction

To increase the accuracy and precision of the acid dissolution technique, the sample should be homogenized prior to analysis. This reduces the grain size of the binder material and releases it from fiber bundles so that it may be dissolved in a shorter time period. Leaving the sample in the acid for a longer period of time to complete the dissolution process can adversely affect the asbestos components, and is not recommended. Homogenization of the sample also ensures that any material removed for analysis will more likely be representative of the entire sample.

Homogenization of friable samples prior to ashing may also accelerate the ashing process; however, the ashing time can simply be increased without affecting the asbestos in the sample. Nonfriable samples, such as vinyl floor tiles, can be broken or shaved into pieces to increase surface area and accelerate the ashing process.

Homogenization and grain size reduction can be accomplished in a variety of ways: 1) hand grinding in a mortar and pestle; 2) crushing with pliers or similar instrument; 3) mixing in a blender; 4) milling (i.e. Wylie mill, cryomill, etc.); or 5) any other technique which seems suitable. If the fibers are extremely long, a pair of scissors or similar implement can be used to reduce the fiber length.

2.3.6 Procedure for Ashing

1) **Weigh appropriate amount of material.**

   There is no restriction on the maximum weight of material used; however, a large amount of material may take longer to ash. Enough material should be used to avoid a significant contribution of weighing errors to the total accuracy and precision.

2) **Place material in crucible, weigh, and cover with lid.**

   Placing a lid on the crucible both minimizes the amount of oxygen available, slowing the rate of combustion of the sample, and prevents any foreign material from falling into the crucible during ashing.

3) **Place crucible into furnace, and ash for at least 6 hours.**

   The furnace temperature at the sample position should be at least 300°C but should not exceed 500°C. If the sample combusts (burns), the temperature of the sample may exceed 500°C. Chrysotile will decompose above approximately 500°C.

   The furnace area should be well-ventilated and the fumes produced by ashing should be exhausted outside the building.

   The ashing time is dependent on the furnace temperature, the amount of sample, and the surface area (grain size). Six hours at 450°C is usually sufficient.

4) **Remove crucible from furnace, allow contents to adjust to room temperature and humidity, and weigh.**

5) **Divide residue weight by starting weight and multiply by 100 to determine weight% residue.**

6) **Analyze residue and/or proceed to acid dissolution procedure.**

If the objective was to remove organic fibers that may be confused optically with asbestos, examine residue with PLM to determine whether any fibers remain.

If the sample is a floor tile, the acid dissolution procedure must now be performed. The residue does not have to be analyzed at this stage.

2.3.7 Use of Solvents for Removal of Organics

Solvent dissolution may be used as a substitute for low temperature ashing for the purpose of removing organic interferences from bulk building materials. However, solvent dissolution, because of the involvement of potentially hazardous reagents such as tetrahydrofuran, amyl acetate, 1-1-1, trichlorethane, etc., requires **that all work be performed with extreme caution inside a biohazard hood.** Material Safety Data Sheets should be reviewed before using any solvent. Solvent dissolution involves more apparatus than does ashing, and requires more time, mainly due to set-up and slow filtration resulting from viscous solvent/residue mixtures.

The following is a brief description of the solvent dissolution process.

1) **Weigh starting material.**

Place approximately 15-25ml of solvent in a 100ml beaker. Add 2.5-3.0 grams (carefully weighed for continued gravimetric tracking) of powdered sample.

2) **Untrasonicate sample.**

Place the beaker in an ultrasonic bath (or ultrasonic stirrer) for approximately 0.5 hours. The sample containers should be covered to preclude escape of an aerosol spray.

3) **Centrifuge sample.**

Weigh centrifuge vial before adding beaker ingredients. Wash beaker with an additional 10-15ml of solvent to remove any remaining concentrate. Then centrifuge

at approximately 2000-2500 rpm for 0.5 hour. Use solvent-resistant centrifuge tubes.

4) **Decant sample, reweigh.**

After separation by centrifuging, decant solvent by pipetting. Leave a small amount of solvent in the centrifuge vial to minimize the risk of decanting solid concentrate. Allow solid concentrate to dry in vial, then reweigh.

2.3.8 Procedure for Acid Dissolution

1) **Weigh starting material, transfer to acid resistant container.**

Small, dry sample weights between 0.1g and 0.5g are recommended (determined for 47mm filters - adjust amount if different diameter filters are used). If too much material is left after acid dissolution the filter can get clogged and prevent complete filtration. Very small samples are also to be avoided, as the weighing errors will have a large effect on the total accuracy and precision of the technique.

2) **Weigh filter.**

3) **Add HCl to sample in container, stir, allow to sit for 2-10 minutes.**

Either concentrated or dilute HCl can be used. If concentrated HCl is used, add enough acid to completely soak the material, allow the reaction to proceed to completion, and then dilute with distilled water. Alternatively, a dilute solution, made by adding concentrated HCl to distilled water, can be used in the place of concentrated HCl. A solution of 1 part concentrated HCl to 3 parts distilled water (approximately 3N solution) has been found to be quite effective in removing components within 5 minutes. For a sample size less than 0.5g, 20-30 ml of a 3N HCl solution is appropriate. In either case (using concentrated or dilute HCl), the reaction will be more effective if the sample has been homogenized first. All obvious signs of reaction (bubbling) should cease before the sample is filtered. Add fresh acid, a ml or two at a time, to ensure complete reaction. It should be noted that if dolomite is present, a 15-20 minute exposure to concentrated HCl may be required to completely dissolve the carbonate materials.

NOTE: Other solvents may be useful for selective dissolution of nonasbestos components. For example, acetic acid will dissolve calcite, and will not dissolve asbestos minerals. If any solvent other than hydrochloric acid is used for the dissolution of inorganic components, the laboratory must be able to demonstrate that the solvent does not remove asbestos from the sample.

4) Filter solution.

Use the pre-weighed filter. Pour the solution into the vacuum filter assembly, then rinse all material from container into filter assembly. Rinse down the inside walls of the glass filter basin and check for particles clinging to the basin after removal.

5) Weigh dried filter + residue, subtract weight of filter from total.

6) Divide residue weight by starting weight and multiply by 100 to determine weight% residue.

7) Analyze residue.

Perform stereomicroscopic examination of residue (can be performed without removing the residue from the filter). Note in particular whether any binder material is still present.

Perform PLM, AEM, or XRD analysis of residue to identify fibers and determine concentration as described in the appropriate sections of this method.

8) Modify procedure if necessary.

If removal of the acid soluble components was not complete, start with a new subsample of material and try any of the following:

a) Decrease grain size of material (by grinding, milling, etc.)
b) Put solutions on hot plate - warm slightly
c) Increase soak time (exercise caution)

9) Calculate relative weight% asbestos in sample.

wt% asbestos in sample = % asbestos in residue x wt% residue ÷ 100

For floor tiles, if the ashing procedure was used first, multiply the weight % of asbestos in the sample, as determined above, by the weight percent of the residue from the ashing procedure, then divide by 100.

Example:
A = wt% residue from ashing = 70%
B = wt% residue from HCl = 20%
C = wt% of asbestos in HCl residue = 50%

wt% asbestos after HCl dissolution = B x C ÷ 100 = 20 x 50 ÷ 100 = 10%

wt% asbestos in floor tile = (B x C ÷ 100) x A ÷ 100 = 10 x 70 ÷ 100 = 7%

If weights are expressed in decimal form, multiply the weight % of asbestos in the sample by the weight % of the residue from the ashing procedure, then multiply by 100.

wt% asbestos after HCl dissolution = B x C = 0.2 x 0.5 = 0.1 (x 100 = 10%)

wt% asbestos in floor tile = (B x C) x A = 0.1 x 0.7 = 0.07 (x 100 = 7%)

2.3.9 Determination of Optimal Precision and Accuracy

The precision of the technique can be determined by extracting multiple subsamples from the original sample and applying the same procedure to each. The optimal accuracy of the technique can be determined by applying gravimetric standards. Mixtures of calcite and asbestos (chrysotile, amosite, etc.) in the following proportions are recommended for testing the accuracy of the acid dissolution technique: 0.1 wt% asbestos/99.9 wt% calcite, 1.0 wt% asbestos/99.0 wt% calcite, and 10 wt% asbestos/90 wt% calcite. Mixtures of cellulose and asbestos are useful for testing the accuracy of the ashing technique.

Mixtures of only two components, as described above, are simplifications of "real-world" samples. The accuracy determined by analyzing these mixtures is considered optimal and may not apply directly to the measurement of each unknown sample. However, analyzing replicates and standards using the full laboratory procedure, including homogenization, ashing, acid dissolution, filtration, and weighing, may uncover steps that introduce significant bias or variation that the laboratory may then correct.

2.3.10 References

1. Kressler, J. R., "Changes in Optical Properties of Chrysotile During Acid Leaching", **The Microscope**, 31, 1983, pp. 165-172.

2. Prentice, J. and M. Keech, "Alteration of Asbestos with Heat", **Microscopy and Analysis**, March 1989.

3. Laughlin, G. and W. C. McCrone, "The Effect of Heat on the Microscopical Properties of Asbestos", **The Microscope**, 37, 1989, pp. 8-15.

2.4 X-Ray Powder Diffraction

2.4.1 Principle and Applicability

The principle of x-ray powder diffraction (XRD) analysis is well established.[1,2] Any solid crystalline material will diffract an incident beam of parallel, monochromatic x-rays whenever Bragg's Law,

$$\lambda = 2d \sin \theta,$$

is satisfied for a particular set of planes in the crystal lattice, where

λ = the x-ray wavelength, Å;
d = the interplanar spacings of the set of reflecting lattice planes, Å and
θ = the angle of incidence between the x-ray beam and the reflecting lattice planes.

By appropriate orientation of a sample relative to the incident x-ray beam, a diffraction pattern can be generated that will be uniquely characteristic of the structure of the crystalline phases present.

Unlike optical methods of analysis, however, XRD cannot determine crystal morphology. Therefore, in asbestos analysis, XRD does not distinguish between fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-6). However, when used in conjunction with methods such as PLM or AEM, XRD techniques can provide a reliable analytical method for the identification and characterization of asbestiform minerals in bulk materials.

For **qualitative** analysis by XRD methods, samples should initially be scanned over limited diagnostic peak regions for the serpentine ($\sim$7.4 Å) and amphibole (8.2-8.5 Å) minerals (Table 2-7). Standard slow-scanning methods for bulk sample analysis may be used for materials shown by PLM to contain significant amounts of asbestos (>5 percent). Detection of minor or trace amounts of asbestos may require special sample preparation and step-scanning analysis. All samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals should be submitted to a full (5°-60° 2θ; 1° 2θ/min) qualitative XRD scan, and their diffraction patterns should be compared with standard reference powder

diffraction patterns[3] to verify initial peak assignments and to identify possible matrix interferences when subsequent quantitative analysis will be performed.

Accurate **quantitative** analysis of asbestos in bulk samples by XRD is critically dependent on particle size distribution, crystallite size, preferred orientation and matrix absorption effects, and comparability of standard reference and sample materials. The most intense diffraction peak that has been shown to be free from interference by prior qualitative XRD analysis should be selected for quantitation of each asbestiform mineral. A "thin-layer" method of analysis[5,6] can be used in which, subsequent to comminution of the bulk material to $\sim 10\ \mu m$ by suitable cryogenic milling techniques, an accurately known amount of the sample is deposited on a silver membrane filter. The mass of asbestiform material is determined by measuring the integrated area of the selected diffraction peak using a step-scanning mode, correcting for matrix absorption effects, and comparing with suitable calibration standards. Alternative "thick-layer" or bulk methods[7,8] are commonly used for **semi-quantitative analysis.**

TABLE 2-6. THE ASBESTOS MINERALS AND THEIR NONASBESTIFORM ANALOGS

Asbestiform	Nonasbestiform	Chemical Abstract Service No.
Serpentine		
Chrysotile	Antigorite, lizardite	12001-29-5
Amphibole		
Anthophyllite asbestos	Anthophyllite	77536-67-5
Cummingtonite-grunerite asbestos (Amosite)	Cummingtonite-grunerite	12172-73-5
Crocidolite	Riebeckite	12001-28-4
Tremolite asbestos	Tremolite	77536-68-6
Actinolite asbestos	Actinolite	77536-66-4

TABLE 2-7. PRINCIPAL LATTICE SPACINGS OF ASBESTIFORM MINERALS[1]

Minerals	Principal d-spacings (Å) and relative intensities			JCPDS Powder diffraction file[2] number
Chrysotile (Serpentine)	7.31_{100} 7.36_{100} 7.10_{100}	3.65_{70} 3.66_{80} 2.33_{80}	4.57_{50} 2.45_{65} 3.55_{70}	21-543[3] 25-645 22-1162 (theoretical)
Amosite (Grunerite)	8.33_{100} 8.22_{100}	3.06_{70} 3.060_{85}	2.756_{70} 3.25_{70}	17-745 (nonfibrous) 27-1170 (UICC)
Anthophyllite	3.05_{100} 3.06_{100}	3.24_{60} 8.33_{70}	8.26_{55} 3.23_{50}	9-455 16-401 (synthetic)
Crocidolite (Riebeckite)	8.35_{100} 8.40_{100}	3.10_{55} 3.12_{55}	2.720_{35} 2.726_{40}	27-1415 (UICC) 19-1061
Actinolite	2.72_{100}	2.54_{100}	3.40_{80}	25-157
Tremolite	8.38_{100} 2.706_{100} 3.13_{100}	3.12_{100} 3.14_{95} 2.706_{60}	2.705_{90} 8.43_{40} 8.44_{40}	13-437[3] 20-1310[3] (synthetic) 23-666 (synthetic mixture w/richterite)

1. This information is intended as a guide only. Complete powder diffraction data, including mineral type and source, should be referred to ensure comparability of sample and reference materials where possible. Additional precision XRD data on amosite, crocidolite, tremolite and chrysotile are available from the U.S. Bureau of Mines, Reference 4.

2. From Reference 3

3. Fibrosity questionable

This XRD method is applicable as a confirmatory method for identification and quantitation of asbestos in bulk material samples that have undergone prior analysis by PLM or other optical methods.

2.4.2 Range and Sensitivity

The range and sensitivity of the method have not been determined. They will be variable and dependent upon many factors, including matrix effects (absorption and interferences), diagnostic reflections selected and their relative intensities, preferred orientation, and instrumental limitations. A detection limit of one percent is feasible given certain sample characteristics.

2.4.3 Limitations

2.4.3.1 Interferences

Since the asbestiform and nonasbestiform analogs of the serpentine and amphibole minerals (Table 2-7) are indistinguishable by XRD techniques unless special sample preparation techniques and instrumentation are used,[9] the presence of nonasbestiform serpentines and amphiboles in a sample will pose severe interference problems in the identification and quantitative analysis of their asbestiform analogs.

The use of XRD for identification and quantitation of asbestiform minerals in bulk samples may also be limited by the presence of other interfering materials in the sample. For naturally-occurring materials, the commonly associated asbestos-related mineral interferences can usually be anticipated. However, for fabricated materials, the nature of the interferences may vary greatly (Table 2-8) and present more serious problems in identification and quantitation.[10] Potential interferences are summarized in Table 2-9 and include the following:

- **Chlorite** has major peaks at 7.19 Å and 3.58 Å that interfere with both the primary (7.31 Å) and secondary (3.65 Å) peaks for serpentine (chrysotile). Resolution of the primary peak to give good quantitative results may be possible when a step-scanning mode of operation is employed.

- **Vermiculite** has secondary peaks at 7.14 Å and 3.56 Å that could interfere with the primary peak (7.31 Å) and a secondary peak (3.65 Å) of serpentine (chrysotile).

TABLE 2-8. COMMON CONSTITUENTS IN BUILDING MATERIAL
(From Ref. 10)

A. Insulation Materials

- Chrysotile
- Amosite
- Crocidolite
- *Rock wool
- *Slag wool
- *Fiber glass
- Gypsum ($CaSO_4 \cdot 2H_20$)
- Vermiculite (micas)
- *Perlite
- Clays (kaolin)
- *Wood pulp
- *Paper fibers (talc, clay carbonate filters)
- Calcium silicates (synthetic)
- Opaques (chromite, magnetite inclusions in serpentine)
- Hematite (inclusions in "amosite")
- Magnesite
- *Diatomaceous earth

B. Flooring Materials

- Calcite
- Dolomite
- Titanium Oxide
- Quartz
- Antigorite
- Chrysotile
- Anthophyllite
- Tremolite
- *Organic binders
- Talc
- Wollastonite

C. Spray Finishes or Paints

- Bassanite
- Carbonate minerals (calcite, dolomite, vaterite)
- Talc
- Tremolite
- Anthophyllite
- Serpentine (including chrysotile)
- Amosite
- Crocidolite
- *Mineral wool
- *Rock wool
- *Slag wool
- *Fiber glass
- Clays (kaolin)
- Micas
- Chlorite
- Gypsum
- Quartz
- *Organic binders and thickeners
- Hydromagnesite
- Wollastonite
- Opaques (chromite, magnetite inclusion in serpentine)
- Hematite (inclusions in "amosite")

D. Cementitious Materials

- Chrysotile
- Amosite
- Crocidolite
- Micas
- Fiber glass
- Cellulose
- Animal hair
- Quartz
- Gypsum
- Calcite
- Dolomite
- Calcium silicates

E. Roofing Materials

- Chrysotile
- Cellulose
- Fiber glass
- Mineral Wool
- Asphalt
- Quartz
- Talc
- Micas

* Amorphous materials--contribute only to overall scattered radiation and increased background radiation.

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TABLE 2-9 INTERFERENCES IN XRD ANALYSIS OF ASBESTIFORM MINERALS

Asbestiform Mineral	Primary diagnostic peaks (approximate d spacings in Å)	Interference
Serpentine Chrysotile	7.3	Nonasbestiform serpentines, (antigorite, lizardite), chlorite, vermiculite, sepiolite, kaolinite, gypsum
	3.7	Nonasbestiform serpentines (antigorite, lizardite), chlorite, vermiculite, halloysite, cellulose
Amphibole Amosite (Grunerite) Anthophyllite Crocidolite (Riebeckite)	3.1	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences, talc, carbonates
Tremolite Actinolite	8.3	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences

- **Sepiolite** produces a peak at 7.47 Å which could interfere with the primary peak (7.31 Å) of serpentine (chrysotile).
- **Halloysite** has a peak at 3.63 Å that interferes with the secondary (3.65 Å) peak for serpentine (chrysotile).
- **Kaolinite** has a major peak at 7.15 Å that may interfere with the primary peak of serpentine (chrysotile) at 7.31 Å when present at concentrations of > 10 percent. However, the secondary serpentine (chrysotile) peak at 3.65 Å may be used for quantitation.
- **Gypsum** has a major peak at 7.5 Å that overlaps the 7.31 Å peak of serpentine (chrysotile) when present as a major sample constituent. This may be removed by careful washing with distilled water, or by heating to 300°C to convert gypsum to plaster of paris (bassanite).
- **Cellulose** has a broad peak that partially overlaps the secondary (3.65 Å) serpentine (chrysotile) peak.[8]

- Overlap of major diagnostic peaks of the amphibole minerals, grunerite (amosite), anthophyllite, riebeckite (crocidolite), and tremolite, at approximately 8.3 Å and 3.1 Å causes mutual interference when these minerals occur in the presence of one another. In some instances adequate resolution may be attained by using step-scanning methods and/or by decreasing the collimator slit width at the x-ray port.

- **Carbonates** may also interfere with quantitative analysis of the amphibole minerals grunerite (amosite), anthophyllite, riebeckite (crocidolite), and tremolite-actinolite. Calcium carbonate ($CaCO_3$) has a peak at 3.035 Å that overlaps major amphibole peaks at approximately 3.1 Å when present in concentrations of >5 percent. Removal of carbonates with a dilute acid wash is possible; however, the time in acid should be no more than 20 minutes to preclude any loss of chrysotile.[11]

- A major talc peak at 3.12 Å interferes with the primary tremolite peak at this same position and with secondary peaks of actinolite (3.14 Å), riebeckite (crocidolite) (3.10 Å), grunerite (amosite) (3.06 Å), and anthophyllite (3.05 Å). In the presence of talc, the major diagnostic peak at approximately 8.3 Å should be used for quantitation of these asbestiform minerals.

The problem of intraspecies and matrix interference is further aggravated by the variability of the silicate mineral powder diffraction patterns themselves, which often makes definitive identification of the asbestos minerals by comparison with standard reference diffraction patterns difficult. This variability results from alterations in the crystal lattice associated with differences in isomorphous substitution and degree of crystallinity. This is especially true for the amphiboles. These minerals exhibit a wide variety of very similar chemical compositions, resulting in diffraction patterns characterized by having major (110) reflections of the monoclinic amphiboles and (210) reflections of orthorhombic anthophyllite separated by less than 0.2 Å.[12]

2.4.3.2 Matrix Effects

If a copper x-ray source is used, the presence of iron at high concentrations in a sample will result in significant x-ray fluorescence, leading to loss of peak intensity, increased background intensity, and an overall decrease in sensitivity. This situation may be corrected by use of an x-ray source other than copper; however, this is often accompanied both by loss of intensity and by decreased resolution of closely spaced reflections. Alternatively, use of a

diffracted beam monochromator will reduce background fluorescent radiation, enabling weaker diffraction peaks to be detected.

X-ray absorption by the sample matrix will result in overall attenuation of the diffracted beam and may seriously interfere with quantitative analysis. Absorption effects may be minimized by using sufficiently "thin" samples for analysis.[5,13,14] However, unless absorption effects are known to be the same for both samples and standards, appropriate corrections should be made by referencing diagnostic peak areas to an internal standard[7,8] or filter substrate (Ag) peak.[5,6]

2.4.3.3 Particle Size Dependence

Because the intensity of diffracted x-radiation is particle-size dependent, it is essential for accurate quantitative analysis that both sample and standard reference materials have similar particle size distributions. The optimum particle size (i.e., fiber length) range for quantitative analysis of asbestos by XRD has been reported to be 1 to 10 μm.[15] Comparability of sample and standard reference material particle size distributions should be verified by optical microscopy (or another suitable method) prior to analysis.

2.4.3.4 Preferred Orientation Effects

Preferred orientation of asbestiform minerals during sample preparation often poses a serious problem in quantitative analysis by XRD. A number of techniques have been developed for reducing preferred orientation effects in "thick layer" samples.[7,8,15] For "thin" samples on membrane filters, the preferred orientation effects seem to be both reproducible and favorable to enhancement of the principal diagnostic reflections of asbestos minerals, actually increasing the overall sensitivity of the method.[12,14] However, further investigation into preferred orientation effects in both thin layer and bulk samples is required.

2.4.3.5 Lack of Suitably Characterized Standard Materials

The problem of obtaining and characterizing suitable reference materials for asbestos analysis is clearly recognized. The National Institute of Standards and Technology can

provide standard reference materials for chrysotile, amosite and crocidolite (SRM 1866) and anthophyllite, tremolite and actinolite (SRM 1867).

In addition, the problem of ensuring the comparability of standard reference and sample materials, particularly regarding crystallite size, particle size distribution, and degree of crystallinity, has yet to be adequately addressed. For example, Langer et al.[18] have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be under-estimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

2.4.4 Precision and Accuracy

Neither the precision nor accuracy of this method has been determined. The individual laboratory should obtain or prepare a set of calibration materials containing a range of asbestos weight percent concentrations in combination with a variety of matrix/binder materials. Calibration curves may be constructed for use in semi-quantitative analysis of bulk materials.

2.4.5 Procedure

2.4.5.1 Sampling

Samples taken for analysis of asbestos content should be collected as specified by EPA[19]

2.4.5.2 Analysis

All samples must be analyzed initially for asbestos content by PLM. XRD may be used as an additional technique, both for identification and quantitation of sample components. **Note: Asbestos is a toxic substance. All handling of dry materials should be performed in a safety-hood.**

2.4.5.2.1 Sample Preparation

The method of sample preparation required for XRD analysis will depend on: (1) the condition of the sample received (sample size, homogeneity, particle size distribution, and overall composition as determined by PLM); and (2) the type of XRD analysis to be performed (qualitative or quantitative; thin-layer or bulk).

Bulk materials are usually received as heterogeneous mixtures of complex composition with very wide particle size distributions. Preparation of a homogeneous, representative sample from asbestos-containing materials is particularly difficult because the fibrous nature of the asbestos minerals inhibits mechanical mixing and stirring, and because milling procedures may cause adverse lattice alterations.

A discussion of specific matrix reduction procedures is given below. Complete methods of sample preparation are detailed in Sections 2.4.5.3 and 2.4.5.4. **Note: All samples should be examined microscopically before and after each matrix reduction step to monitor changes in sample particle size distribution, composition, and crystallinity, and to ensure sample representativeness and homogeneity for analysis.**

2.4.5.2.2 Milling

Mechanical milling of asbestos materials has been shown to decrease fiber crystallinity, with a resultant decrease in diffraction intensity of the specimen; the degree of lattice alteration is related to the duration and type of milling process.[20-23] Therefore, all milling times should be kept to a minimum.

For **qualitative analysis**, particle size is not usually of critical importance and initial characterization of the material with a minimum of matrix reduction is often desirable to document the composition of the sample as received. Bulk samples of very large particle size (>2-3 mm) should be comminuted to ~ 100 μm. A mortar and pestle can sometimes be used in size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced by cutting with a razor blade in a mortar, or by grinding in a suitable mill (e.g., a microhammer mill or equivalent). When using a mortar for grinding or cutting, the sample should be moistened with ethanol, or some other

suitable wetting agent, to minimize exposure, and the procedure should be performed in a HEPA-filtered hood.

For accurate, reproducible **quantitative analysis**, the particle size of both sample and standard materials should be reduced to $\sim 10\ \mu m$. Dry ball milling at liquid nitrogen temperatures (e.g., Spex Freezer Mill®, or equivalent) for a maximum time of 10 minutes (some samples may require much shorter milling time) is recommended to obtain satisfactory particle size distributions while protecting the integrity of the crystal lattice.[5] Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to $< 10\ \mu m$.[8,16]

Final particle size distributions should always be verified by optical microscopy or another suitable method.

2.4.5.2.3 Ashing

For materials shown by PLM to contain large amounts of cellulose or other organic materials, it may be desirable to ash prior to analysis to reduce background radiation or matrix interference. Since chrysotile undergoes dehydroxylation at temperatures between 550°C and 650°C, with subsequent transformation to forsterite,[24,25] ashing temperatures should be kept below 500°C. Use of a muffle furnace is recommended. In all cases, calibration of the furnace is essential to ensure that a maximum ashing temperature of 500°C is not exceeded (see Section 2.3).

2.4.5.2.4 Acid Washing

Because of the interference caused by gypsum and some carbonates in the detection of asbestiform minerals by XRD (see Section 2.4.3.1), it may be necessary to remove these interferences by a simple acid washing procedure prior to analysis (see Section 2.3).

2.4.5.3 Qualitative Analysis

2.4.5.3.1 Initial Screening of Bulk Material

Qualitative analysis should be performed on a representative, homogeneous portion of the sample, with a minimum of sample treatment, using the following procedure:

1. Grind and mix the sample with a mortar and pestle (or equivalent method, see Section 2.4.5.2.2) to a final particle size sufficiently small ($\sim 100\ \mu m$) to allow adequate packing into a sample holder.

2. Pack sample into a standard bulk sample holder. Care should be taken to ensure that a representative portion of the milled sample is selected for analysis. Particular care should be taken to avoid possible size segregation of the sample. **(Note: Use of back-packing method[26] for bulk sample preparation may reduce preferred orientation effects.)**

3. Mount the sample on the diffractometer and scan over the diagnostic peak regions for the serpentine (~ 7.4 Å) and amphibole (8.2-8.5 Å) minerals (see Table 2-7). The x-ray diffraction equipment should be optimized for intensity. A slow scanning speed of 1° 2θ/min is recommended for adequate resolution. Use of a sample spinner is recommended.

4. Submit all samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals to a full qualitative XRD scan (5°-60° 2θ; 1° 2θ/min) to verify initial peak assignments and to identify potential matrix interferences when subsequent quantitative analysis is to be performed.

5. Compare the sample XRD pattern with standard reference powder diffraction patterns (i.e., JCPDS powder diffraction data[3] or those of other well-characterized reference materials). Principal lattice spacings of asbestiform minerals are given in Table 2-7; common constituents of bulk insulation and wall materials are listed in Table 2-8.

2.4.5.3.2 Detection of Minor or Trace Constituents

Routine screening of bulk materials by XRD may fail to detect small concentrations ($< 1\%$) of asbestos. The limits of detection will, in general, be improved if matrix absorption effects are minimized, and if the sample particle size is reduced to the optimal 1 to 10 μm range, provided that the crystal lattice is not degraded in the milling process. Therefore, in those instances when confirmation of the presence of an asbestiform mineral at very low levels is required, or where a negative result from initial screening of the bulk material by XRD (see Section 2.4.5.3.1) is in conflict with previous PLM results, it may be desirable to prepare the sample as described for quantitative analysis (see Section 2.4.5.4) and step-scan over appropriate 2θ ranges of selected diagnostic peaks (Table 2-7). Accurate

transfer of the sample to the silver membrane filter is not necessary unless subsequent quantitative analysis is to be performed.

2.4.5.4 Quantitative Analysis

The proposed method for quantitation of asbestos in bulk samples is a modification of the NIOSH-recommended thin-layer method for chrysotile in air.[6] A thick-layer bulk method involving pelletizing the sample may be used for semi-quantitative analysis;[7,8] however, this method requires the addition of an internal standard, use of a specially fabricated sample press, and relatively large amounts of standard reference materials. Additional research is required to evaluate the comparability of thin- and thick-layer methods for quantitative asbestos analysis.

For quantitative analysis by thin-layer methods, the following procedure is recommended:

1. Mill and size all or a substantial representative portion of the sample as outlined in Section 2.4.5.2.2.

2. Dry at 60°C for 2 hours; cool in a desiccator.

3. Weigh accurately to the nearest 0.01 mg.

4. Samples shown by PLM to contain large amounts of cellulosic or other organic materials, gypsum, or carbonates, should be submitted to appropriate matrix reduction procedures described in Sections 2.4.5.2.3 and 2.4.5.2.4. After ashing and/or acid treatment, repeat the drying and weighing procedures described above, and determine the percent weight loss, L.

5. Quantitatively transfer an accurately weighed amount (50-100 mg) of the sample to a 1-L volumetric flask containing approximately 200 mL isopropanol to which 3 to 4 drops of surfactant have been added.

6. Ultrasonicate for 10 minutes at a power density of approximately 0.1 W/mL, to disperse the sample material.

7. Dilute to volume with isopropanol.

8. Place flask on a magnetic-stirring plate. Stir.

9. Place silver membrane filter on the filtration apparatus, apply a vacuum, and attach the reservoir. Release the vacuum and add several milliliters of isopropanol to the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw

an aliquot from the center of the suspension so that total sample weight, W_T, on the filter will be approximately 1 mg. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and repeat the procedure with a clean pipet. Transfer the aliquot to the reservoir. Filter rapidly under vacuum. Do not wash the reservoir walls. Leave the filter apparatus under vacuum until dry. Remove the reservoir, release the vacuum, and remove the filter with forceps. (Note: Water-soluble matrix interferences such as gypsum may be removed at this time by careful washing of the filtrate with distilled water. Extreme care should be taken not to disturb the sample.)

10. Attach the filter to a flat holder with a suitable adhesive and place on the diffractometer. Use of a sample spinner is recommended.

11. For each asbestos mineral to be quantitated, select a reflection (or reflections) that has (have) been shown to be free from interferences by prior PLM or qualitative XRD analysis and that can be used unambiguously as an index of the amount of material present in the sample (see Table 2-7).

12. Analyze the selected diagnostic reflection(s) by step-scanning in increments of 0.02° 2θ for an appropriate fixed time and integrating the counts. (A fixed count scan may be used alternatively; however, the method chosen should be used consistently for all samples and standards.) An appropriate scanning interval should be selected for each peak, and background corrections made. For a fixed time scan, measure the background on each side of the peak for one-half the peak-scanning time. The net intensity, I_s, is the difference between the peak integrated count and the total background count.

13. Determine the net count, I_{Ag}, of the filter 2.36 Å silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak, I°_{Ag}. Scan times may be less for measurement of silver peaks than for sample peaks; however, they should be constant throughout the analysis.

14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 Å peak of an α-quartz reference crystal). After each unknown is scanned, determine the net count, I°_r, of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by I°_r:

$$\hat{I}_a = \frac{I_a}{I_r^\circ}, \quad \hat{I}_{Ag} = \frac{I_{Ag}}{I_r^\circ}, \text{ and } \quad \hat{I}_{Ag}^\circ = \frac{I_{Ag}^\circ}{I_r^\circ}$$

2.4.6 Calibration

2.4.6.1 Preparation of Calibration Standards

1. Mill and size standard asbestos materials according to the procedure outlined in Section 2.4.5.2.2. **Equivalent standardized matrix reduction and sizing techniques should be used for both standard and sample materials.**

2. Dry at 100°C for 2 hours; cool in a desiccator.

3. Prepare two suspensions of each standard in isopropanol by weighing approximately 10 and 50 mg of the dry material to the nearest 0.01 mg. Transfer each to a 1-L volumetric flask containing approximately 200 mL isopropanol to which a few drops of surfactant have been added.

4. Ultrasonicate for 10 minutes at a power density of approximately 0.1 W/mL, to disperse the asbestos material.

5. Dilute to volume with isopropanol.

6. Place the flask on a magnetic stirring plate. Stir.

7. Prepare, in triplicate, a series of at least five standard filters to cover the desired analytical range, using appropriate aliquots of the 10 and 50 mg/L suspensions. For each standard, mount a silver membrane filter on the filtration apparatus. Place a few mL of isopropanol in the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Keep the tip of the pipet near the surface of the isopropanol. Filter rapidly under vacuum. Do not wash the sides of the reservoir. Leave the vacuum on for a time sufficient to dry the filter. Release the vacuum and remove the filter with forceps.

2.4.6.2 Analysis of Calibration Standards

1. Mount each filter on a flat holder. Perform step scans on selected diagnostic reflections of the standards and reference specimen using the procedure outlined in Section 2.4.5.4, step 12, and the same conditions as those used for the samples.

2. Determine the normalized intensity for each peak measured, $\hat{I}^{\circ}_{std}$, as outlined in Section 2.4.5.4, step 14.

2.4.7 Calculations

For each asbestos reference material, calculate the exact weight deposited on each standard filter from the concentrations of the standard suspensions and aliquot volumes. Record the weight, w, of each standard. Prepare a calibration curve by regressing $\hat{I}^{\circ}_{std}$, on w. Poor reproducibility ($\pm$15 percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight-line equation.

Determine the slope, m, of the calibration curve in counts/microgram. The intercept, b, of the line with the $\hat{I}^{\circ}_{std}$ axis should be approximately zero. A large negative intercept indicates an error in determining the background. This may arise from incorrectly measuring the baseline or from interference by another phase at the angle of background measurement. A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

Using the normalized intensity, $\hat{I}_{Ag}$ for the attenuated silver peak of a sample, and the corresponding normalized intensity from the unattenuated silver peak $\hat{I}^{\circ}_{Ag}$, of the sample filter, calculate the transmittance, T, for each sample as follows:[27,28]

$$T = \frac{\hat{I}_{Ag}}{\hat{I}^{\circ}_{Ag}}$$

Determine the correction factor, f(T), for each sample according to the formula:

$$f(T) = \frac{-R(\ln T)}{1 - T^R}$$

where

$$R = \frac{\sin \theta_{Ag}}{\sin \theta_a}$$

θ_{Ag} = angular position of the measured silver peak (from Bragg's Law), and

θ_a = angular position of the diagnostic asbestos peak.

Calculate the weight, W_a, in micrograms, of the asbestos material analyzed for in each sample, using the absorption corrections:

$$W_a = \frac{\hat{I}_a f(t) - b}{m}$$

Calculate the percent composition, P_a, of each asbestos mineral analyzed for in the parent material, from the total sample weight, W_T, on the filter:

$$P_a = \frac{W_a(1 - .01L)}{W_T} \times 100$$

where

P_a = percent asbestos mineral in parent material;

W_a = mass of asbestos mineral on filter, in μg;

W_T = total sample weight on filter, in μg;

L = percent weight loss of parent material on ashing and/or acid treatment (see Section 2.4.5.4).

2.4.8 References

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2.5 Analytical Electron Microscopy

2.5.1 Applicability

Analytical electron microscopy (AEM) can often be a reliable method for the detection and positive identification of asbestos in some bulk building materials, both friable and nonfriable. The method is particularly applicable to bulk materials that contain a large amount of interfering materials that can be removed by ashing and/or dissolution and contain asbestos fibers that are not resolved by PLM techniques. Many floor tiles and plasters would be included in this type of sample. In combination with suitable specimen preparation techniques, the AEM method can also be used to quantify asbestos concentrations.

2.5.2 Range

The range is dependent on the type of bulk material being analyzed. The upper detection limit is 100%, and the lower detection limit can be as low as 0.0001% depending on the extent to which interfering materials can be separated during the preparation of AEM

specimens, the sophistication of the AEM preparation, and the amount of labor expended on AEM examination.

2.5.3 Interferences

The presence of a large amount of binder/matrix materials associated with fibers can make it difficult to positively identify fibers as asbestos. The portion of the fiber examined by either electron diffraction or energy dispersive x-ray analysis (EDXA) must be free of binder/matrix materials.

2.5.4 Precision and Accuracy

The precision and accuracy of the method have not been determined.

2.5.5 Procedures

The procedures for AEM specimen preparation depend on the data required. In analysis of floor tiles, the weighed residue after removal of the matrix components (see Section 2.3, Gravimetry) is often mostly asbestos, and the task is primarily to identify the fibers. In this situation the proportion of asbestos in the residue can be estimated by AEM and this estimate can be used to refine the gravimetric result. For many floor tiles, the final result is not very sensitive to errors in this estimation because the proportion of asbestos in the residue is very high. For samples in which this is not the case, precise measurements can be made using a quantitative AEM preparation, in which each grid opening of the specimen grid corresponds to a known weight of the original sample or of a concentrate derived from the original sample. Asbestos fibers on these grids are then identified and measured, using a fiber counting protocol which is directed towards a precise determination of mass concentration. This latter procedure is suitable for samples of low asbestos concentration, or for those in which it is not possible to remove a large proportion of the matrix material.

2.5.5.1 AEM Specimen Preparation for Semi-Quantitative Evaluation

The residual material from any ashing or dissolution procedures (see Section 2.3) used (usually trapped on a membrane filter) should be placed in a small volume of ethanol or another solvent such as acetone or isopropyl alcohol, in a disposable beaker, and dispersed

by treatment in an ultrasonic bath. A small volume of this suspension (approximately 3μl) should be pipetted onto the top of a carbon-coated TEM grid. The suspension should be allowed to dry under a heat lamp. The grid is then ready for examination.

Samples that are not conducive to ashing or dissolution may also be prepared in this way for AEM analysis. A few milligrams of the sample may be ground in a mortar and pestle or milled, dispersed in ethanol or another solvent using an ultrasonic bath, and pipetted onto a grid as described previously.

2.5.5.2 AEM Specimen Preparation for Quantitative Evaluation

The objective of this preparation is to obtain a TEM grid on which a known weight of the bulk sample is represented by a known area of the TEM grid. A known weight of the bulk sample, or of the residue after extraction, should be dispersed in a known volume of distilled water. Aliquots of this dispersion should then be filtered through 0.22 μm pore-size MCE or 0.2 μm pore-size PC filters, using filtration techniques as described for analysis of water samples.[1] In order to obtain filters of appropriate particulate loading for AEM analysis, it may be necessary to perform serial dilutions of the initial dispersion. TEM grids should then be prepared from appropriately-loaded filters, using the standard methods.[2]

Determination of the mass concentration of asbestos on the TEM grids requires a different fiber counting protocol than that usually used for determination of numerical fiber concentrations. Initially, the grids should be scanned to determine the dimensions of the largest asbestos fiber or fiber bundle on the specimens. The volume of this fiber or bundle should be calculated. The magnification of the AEM should be set at a value for which the length of this fiber or bundle just fills the fluorescent screen. Asbestos fiber counting should then be continued at this magnification. The count should be terminated when the volume of the initial large fiber or bundle represents less than about 5% of the integrated volume of all asbestos fibers detected. This counting strategy ensures that the fiber counting effort is directed toward those fibers which contribute most to the mass, and permits a precise mass concentration value to be obtained.

2.5.5.2.1 Identification

To document the positive identification of asbestos in a sample, the analyst should record the following physical properties: morphology data, electron diffraction data, EDXA data, and any other distinguishing characteristics observed. For fibrous structures identified as nonasbestos, the unique physical property or properties that differentiate the material from asbestos should be recorded.

The purpose of the identification data collected is to prevent or limit false negatives and false positives. This can be accomplished by having a system for measuring and recording the d-spacings and symmetry of the diffraction patterns, determining the relative abundance of the elements detected by EDXA, and comparing these results to reference data. The laboratory should have a set of reference asbestos materials from which a set of reference diffraction patterns and x-ray spectra have been developed. Also, the laboratory should have available reference data on the crystallography and chemical composition of minerals that might analytically interfere with asbestos.

2.5.6 References

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2.6 Other Methodologies

Additional analytical methods (e.g. Scanning Electron Microscopy) may be applicable for some bulk materials. However, the analyst should take care to recognize the limitations of any analytical method chosen. Conventional SEM, for example, cannot detect small diameter fibers ($\sim < 0.2\mu m$), and cannot determine crystal structure. It is, however, very useful for observing surface features in complex particle matrices, and for determining elemental compositions.